Operator's Handbook

LLT Series

Liquid Helium Transfer Tubes

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OXFORD INSTRUMENTS

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Warnings

Before you attempt to install or operate this equipment for the first time, please make sure that you are aware of the precautions that you must take to ensure your own safety. The booklet *Safety Matters* covers the hazards that you are likely to encounter when using cryogens.

Please read this manual carefully before assembling or commissioning the system. It is possible to damage the system beyond repair if the correct procedures are not followed.

Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in this manual. The warranty may be affected if the system is misused, or the recommendations in this handbook are not followed.

1 Introduction

1.1 Introduction to this manual

The LLT liquid helium transfer tube is a gas flow shielded, vacuum insulated tube designed for use with a continuous flow cryostat. The exhaust gas from the cryostat is used to cool a radiation shield around the liquid delivery tube, reducing the heat load on the liquid helium.

Eight versions are available. The LLT600 and LLT650 are used with cryostats which have horizontal or nearly horizontal entry arms. The LLT700 and LLT750 are used for vertical entry arms. The LLT600/10 has a 1.0 m dewar leg; the LLT600/13 has a 1.3 m dewar leg, and so on for the other versions. All of them have flexible sections so that the cryostat can move relative to the storage dewar.

A needle valve is fitted in the leg that fits in the storage dewar. This is used to control the flow of liquid helium through the transfer tube. LLT650 and LLT750 are fitted with stepper motors for automatic or remote operation of the needle valve. They can be used with Oxford Instruments ITC temperature controllers to adjust the liquid helium flow rate automatically; (further details are given in the Operators Handbook for Auto needle valves and Auto LLT). LLT 600 and LLT 700 are used for manual operation, and are illustrated below.

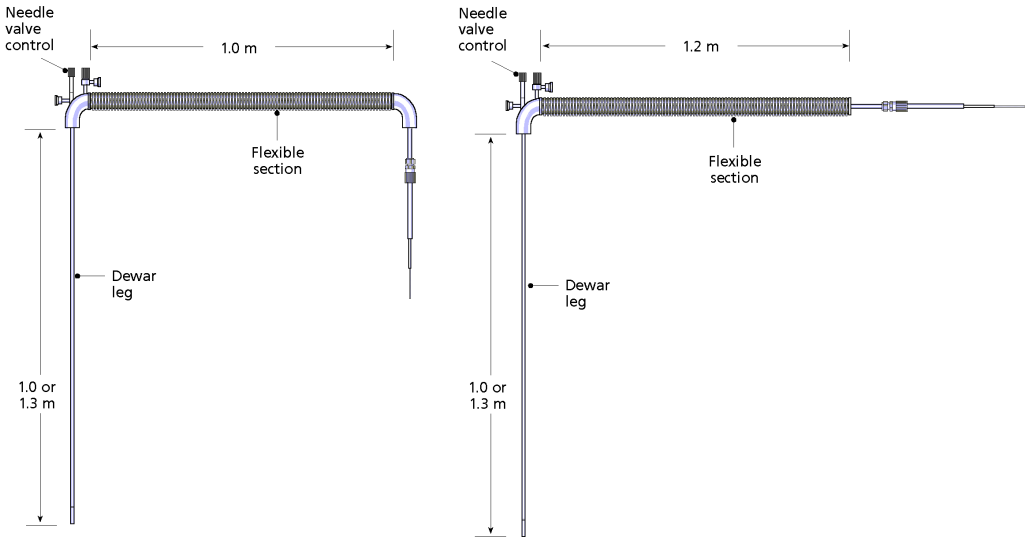

Figure 1 LLT transfer tube options (manual needle valve)

1.2 Safety requirements

Please refer to the separate safety booklet *Safety Matters* which should have been supplied with the system. This includes information about the properties of liquid nitrogen and liquid helium, and detailed recommendations about the precautions you should take. It is your responsibility to ensure your own safety, and the safety of the people working around you.

2 Unpacking and preparation

2.1 Unpacking the transfer tube

Carefully remove the transfer tube from the packing and inspect it to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford Instruments immediately.

2.2 Preparing the transfer tube for operation

2.2.1 Evacuating the vacuum space

The vacuum space has to be pumped to high vacuum to make sure that it gives the required thermal insulation. When the transfer tube is new all the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This does not mean that the transfer tube is leaking, just that the new materials are being cleaned by the vacuum. The transfer tube has been pumped to high vacuum and baked before it left the factory. However, the vacuum space should be pumped occasionally, especially when the transfer tube is new.

If water condenses on the outside of the transfer tube while it is being used, or if the cryostat does not reach base temperature or needs a high flow rate to reach base temperature, this may indicate that the transfer tube vacuum space needs to be pumped again.

The LLT transfer tube is fitted with a vacuum valve with an integral NW16 flange.

Connect the pumping system to the NW16 flange on the valve. We recommend that you use a diffusion pump or turbo-molecular pump. If a rotary pump is used to back the high vacuum pump, the gas ballast valve should be left closed unless there is reason to believe that the transfer tube is badly contaminated with water vapour.

Evacuate the pumping line to a medium vacuum with the rotary pump before you open the valve. Open the valve by turning the knob anti-clockwise a few turns, and pump the vacuum space to a high vacuum. Leave it to pump for at least one hour, and preferably overnight. Close the valve firmly before you vent the pumping line and remove the pumping system.

2.2.2 Protective cover

All LLT transfer tubes are supplied with a protective cover to prevent damage to the delicate part that fits into the cryostat arm. Unscrew and remove this cover before you use the transfer tube, but keep it in a safe place for future use.

3 Using the transfer tube

The cryostat manual explains how to use the LLT transfer tube in detail. The following information will only be needed if you have problems running the system.

3.1 Adjusting the cryostat entry arm lock nuts

If the transfer tube has been supplied as part of a system there should be no need to adjust the lock nut. Normally you only need to follow this procedure if the transfer tube has to be adjusted to fit a different cryostat. The components are labelled in Figure 2. The nut and lock nuts are used to compress a collet, so that the assembly is firmly fixed to the transfer tube arm.

a) Hold the nut and loosen the lock nut using two spanners so that the assembly can slide freely along the transfer tube delivery arm.

b) Push the transfer tube delivery arm fully into the cryostat arm, so that the PTFE seal meets the mating surface inside the cryostat. (Some cryostats do not have entry arms and no PTFE seal is required. Push the transfer tube in as far as it will go.)

c) Screw the knurled nut fully onto the thread on the entry arm, and then unscrew it by two or three turns.

d) Gently push the transfer tube delivery arm into the cryostat to make sure that the PTFE seal is seating (or the transfer tube is as far as possible into the cryostat) while you tighten the lock nut onto the transfer tube again.

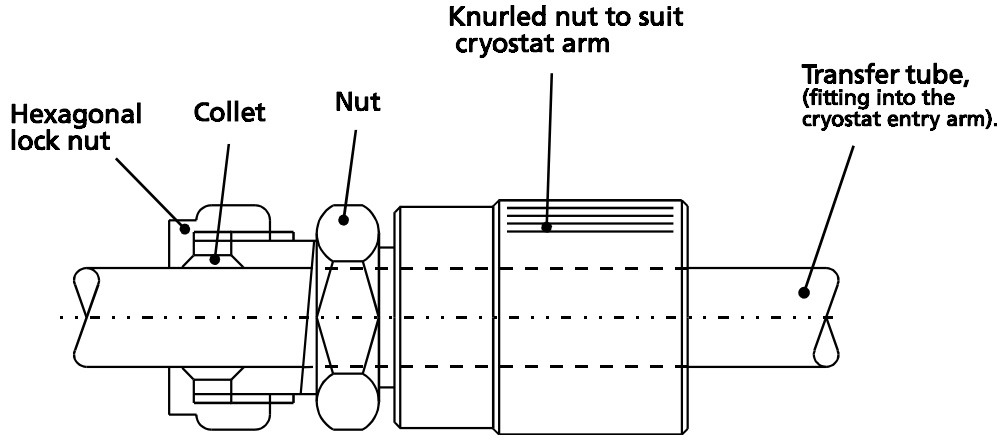

Figure 2 Adjusting the cryostat entry arm nut

3.2 Cooling down the transfer tube

If the transfer tube has been supplied as part of a system please refer to the cryostat manual for operating instructions. If it has been supplied separately the following instructions explain how to cool it down safely.

Close the needle valve on the transfer tube fully by rotating the knurled nut clockwise (looking from above) or setting a gas flow of 0% on the temperature controller. Then open it a maximum of 4 turns or set the gas flow to 100%.

Check that the PTFE seal on the end of the transfer tube is clean and undamaged (if it is required). There should be no grease on it.

Open the needle valve on the VC31 fully, by turning it anti-clockwise. Switch on the GF3 pump.

Warning **Make sure that you have taken the necessary precautions to ensure your own safety and the safety of other people working near you.**

Slowly lower the dewar leg of the transfer tube into the liquid helium. Some liquid will be used to cool the leg, and the dewar exhaust must be open to allow this gas to escape. If you try to cool the leg too quickly a large amount of liquid will be wasted, and you could be burnt by the cold gas.

As soon as the dewar leg has been loaded into the liquid helium, push the other end into the entry arm of the cryostat until the knurled nut just touches the thread on the arm. Do not engage the thread yet. Connect the exhaust gas line, and watch the flow gauge on the VC31. This flow should increase gradually as the transfer tube cools. After a few minutes when the flow is about 1.5 litres per hour, engage the nut on the transfer tube onto the thread on the cryostat arm and tighten it. The flow will drop initially, then rise again. If the flow does not reach about 1.5 litres hour after 20 minutes, the transfer tube may be blocked, or the needle valve may not be opening correctly. It is good practice to fit a soccer ball bladder to the exhaust of the storage dewar and squeeze and release it occasionally to keep the pressure slightly above atmospheric pressure, to prevent air getting inside the storage dewar.

If you have persistent problems with blockages in the transfer tube you may be able to reduce the risk by altering the procedure as follows. Before you start to lower the transfer tube leg into the storage dewar, connect the " From Cryostat" connection of the VC31 to the cryostat end of the transfer tube using a short length of rubber tube. Use the GF3 pump and VC31 to draw helium gas through the transfer tube until it is cold and then, wearing thick gloves, quickly remove the rubber tube and insert the transfer tube into the cryostat arm as described above. Connect the VC31 "From Cryostat" tube to the normal exhaust port and continue as described in the next paragraph.

The flow rate will then drop again because of the impedance of the small tube in the cryostat (which has not yet cooled down). As this tube cools, the flow will increase again, and after about 10 minutes it should typically increase to 1.5 litres per hour. If not, there may be a blockage in the cryostat.

The cryostat heat exchanger and sample should now be cooling steadily, and the transfer tube and cryostat arm may contract by different amounts. The knurled nut on the cryostat arm should be tightened again occasionally, to make sure that it maintains the seal in the cryostat, so that the liquid helium does not by-pass the cryostat.

3.3 Clearing blockages in the transfer tube

If the transfer tube becomes blocked while you are using it, remove it from the cryostat and the storage dewar with the needle valve open. Connect a supply of clean helium gas to the delivery arm end of the transfer tube with rubber tube and pressurise it to about 500 mbar. Allow the transfer tube to warm up, and if necessary warm it slightly with a hot air blower. This is unlikely to affect the inner tube very much because of the vacuum insulation, but it does warm the ends of the tube and the needle valve and it is likely to help clear the blockage. Make sure that you can feel gas escaping through the inlet hole in the dewar leg.

Warning: **In an extreme case there may be a pressure rise, which blows the rubber tube from the transfer tube. Do not hold the transfer tube unless you are wearing the correct protective clothing and a facemask to prevent cold burns.**